

CLAIMS

1.- Process for the production and purification of lactide, characterized in that, starting from an aqueous solution of lactic acid or of lactic acid derivatives, the stages comprise:

- a) evaporation of the free water and of a portion of the water of constitution until oligomers having a molecular mass of between 400 and 2000 amu, a total acidity as lactic acid equivalent of between 119 and 124.5% and an optical purity, expressed as L-lactic acid, of between 90 and 100% are obtained;
- b) feeding the mixture comprising a depolymerization catalyst and the oligomers obtained in a) to a depolymerization reactor with production of:
  - b1) a lactide-rich vapour phase, and
  - b2) an oligomer-rich liquid residue;
- c) selective condensation of the lactide-rich vapour (b1) with recovery, in the liquid form, of a crude lactide product freed from the volatile compounds;
- d) melt crystallization of the crude lactide product (c), with production of a purified lactide fraction having a residual acidity of less than 10 meq/kg, a water content of less than 200 ppm and a meso-lactide content of less than 1%;
- e) aqueous treatment of the residual fractions from the melt crystallization, consisting of:
  - e1) extractive and controlled crystallization of these fractions in an aqueous medium, with control of the geometry of the crystals formed and with segregation of the lactide suspension towards the solid phase and of the impurities

towards the liquid phase, so as to carry out aqueous extraction of the impurities;

e2) separation of the suspension of crystals (e1) from the liquid phase and then draining, which separates a wet cake rich in lactide crystals from a liquid phase depleted in lactide and laden with impurities;

e3) drying the wet cake (e2), which provides the prepurified lactide.

2.- Process for the production and purification of lactide, characterized in that, starting from an aqueous solution of lactic acid or of lactic acid derivatives, the stages comprise:

a) evaporation of the free water and of a portion of the water of constitution until oligomers having a molecular mass of between 400 and 2000 amu, a total acidity as lactic acid equivalent of between 119 and 124.5% and an optical purity, expressed as L-lactic acid, of between 90 and 100% are obtained;

b) feeding the mixture comprising a depolymerization catalyst and the oligomers obtained in a) to a depolymerization reactor with production of:

b1) a lactide-rich vapour phase, and

b2) an oligomer-rich liquid residue;

c) selective condensation of the lactide-rich vapour (b1) with recovery, in the liquid form, of a crude lactide product freed from the volatile compounds;

d) aqueous treatment of the crude lactide product resulting from (c) consisting of:

d1) extractive and controlled crystallization in an aqueous medium, with control of the geometry of the crystals formed and with segregation of the

lactide suspension towards the solid phase and of the impurities towards the liquid phase, so as to carry out aqueous extraction of the impurities;

5       d2) separation of the suspension of crystals (d1) from the liquid phase and then draining, which separates a wet cake rich in lactide crystals from a liquid phase depleted in lactide and laden with impurities;

10       d3) drying the wet cake (d2), which provides a prepurified lactide;

      e) melt crystallization of the prepurified lactide (d3), with production of a purified lactide fraction having a residual acidity of less than 10 meq/kg, a water content of less than 200 ppm and a meso-lactide  
15       content of less than 1%.

3.- Process for the production of polylactide, characterized in that the stages of production and of purification of lactide, starting from an aqueous solution of lactic acid or of lactic acid derivatives,  
20       comprise:

      a) evaporation of the free water and of a portion of the water of constitution until oligomers having a molecular mass of between 400 and 2000 amu, a total acidity as lactic acid equivalent of between 119 and  
25       124.5% and an optical purity, expressed as L-lactic acid, of between 90 and 100% are obtained;

      b) feeding the mixture comprising a depolymerization catalyst and the oligomers obtained in a) to a depolymerization reactor with production of:

30       b1) a lactide-rich vapour phase, and

      b2) an oligomer-rich liquid residue;

      c) selective condensation of the lactide-rich vapour (b1)

with recovery, in the liquid form, of a crude lactide product freed from the volatile compounds;

d) melt crystallization of the crude lactide product (c), with production of a purified lactide fraction having a residual acidity of less than 10 meq/kg, a water content of less than 200 ppm and a meso-lactide content of less than 1%;

e) aqueous treatment of the residual fractions from the melt crystallization, consisting of:

10 e1) extractive and controlled crystallization of these fractions in an aqueous medium, with control of the geometry of the crystals formed and with segregation of the lactide suspension towards the solid phase and of the impurities towards the liquid phase, so as to carry out aqueous extraction of the impurities;

15 e2) separation of the suspension of crystals (e1) from the liquid phase and then draining, which separates a wet cake rich in lactide crystals from a liquid phase depleted in lactide and laden with impurities;

20 e3) drying the wet cake (e2), which provides the prepurified lactide;

f) polymerization of lactide to polylactide.

25 4.- Process for the production of polylactide, characterized in that the stages of production and of purification of lactide, starting from an aqueous solution of lactic acid or of lactic acid derivatives, comprise:

30 a) evaporation of the free water and of a portion of the water of constitution until oligomers having a molecular mass of between 400 and 2000 amu, a total

acidity as lactic acid equivalent of between 119 and 124.5% and an optical purity, expressed as L-lactic acid, of between 90 and 100% are obtained;

- b) feeding the mixture comprising a depolymerization catalyst and the oligomers obtained in a) to a depolymerization reactor with production of:
  - b1) a lactide-rich vapour phase, and
  - b2) an oligomer-rich liquid residue;
- c) selective condensation of the lactide-rich vapour (b1) with recovery, in the liquid form, of a crude lactide product freed from the volatile compounds;
- d) aqueous treatment of the crude lactide product resulting from (c) consisting of:
  - d1) extractive and controlled crystallization in an aqueous medium, with control of the geometry of the crystals formed and with segregation of the lactide suspension towards the solid phase and of the impurities towards the liquid phase, so as to carry out aqueous extraction of the impurities;
  - d2) separation of the suspension of crystals (d1) from the liquid phase and then draining, which separates a wet cake rich in lactide crystals from a liquid phase depleted in lactide and laden with impurities;
  - d3) drying the wet cake (d2), which provides a prepurified lactide;
- e) melt crystallization of the prepurified lactide (d3), with production of a purified lactide fraction having a residual acidity of less than 10 meq/kg, a water content of less than 200 ppm and a meso-lactide content of less than 1%;
- f) polymerization of lactide to polylactide.

5.- Process according to any one of the preceding claims, characterized in that the starting lactic acid derivatives comprise lactic acid esters.

6.- Process according to one of Claims 1 to 4, characterized in that the starting lactic acid derivatives comprise a mixture of lactic acid and of one or more lactic acid esters.

7.- Process according to any one of the preceding claims, characterized in that the crude lactide product is enriched in prepurified lactide fractions originating from the aqueous treatment of the residual fractions from the melt crystallization.

8.- Process according to any one of the preceding claims, characterized in that the prepurified lactide resulting from the aqueous treatment can be recycled at any point in the production of purified lactide.

9.- Process according to any one of the preceding claims, characterized in that the content of D-lactide during the progression of the process is controlled by polymerization by ring opening of the prepurified lactide.

10.- Process according to any one of the preceding claims, characterized in that the prepurified lactide exhibits a residual water content of between 50 and 1000 ppm, a total lactide content of between 70 and 99%, a content of lactic acid and lactic acid oligomers of between 0 and 5% and a meso-lactide content of between 0 and 15%.

11.- Process for the production of polylactide according to Claim 3 or 4, characterized in that the polymerization of the purified and/or prepurified lactide

comprises the stages:

- a) of addition of a catalyst or mixture of catalysts to the lactide;
- b) of initiation of the prepolymerization with addition, to the mixture (a), of optional comonomers, of oligomers, of prepolymers, of stabilizers, of fillers, of reinforcing agents or of polymerization moderators;
- c) of polymerization in an extruder with addition of optional comonomers, of oligomers, of prepolymers, of stabilizers, of fillers, of reinforcing agents or of polymerization moderators.

12.- Process for the production of polylactide according to Claim 3 or 4, characterized in that the polymerization of the purified and/or prepurified lactide comprises the stages:

- a) of addition of a catalyst or a mixture of catalysts to the lactide;
- b) of polymerization in an extruder with addition, to the mixture (a), of optional comonomers, of oligomers, of prepolymers, of stabilizers, of fillers, of reinforcing agents or of polymerization moderators.

13.- Process for the production of polylactide according to Claims 3, 4, 11 or 12, characterized in that, during the purification and the production of polylactide, the recycled fractions of lactic acid or of its derivatives are introduced in the purification stage of the process for the production of lactic acid or of its derivatives.

14.- Process for the production of lactide according to Claim 1 or 2, characterized in that, during the production and the purification of lactide, the recycled fractions of lactic acid or of its derivatives

are introduced in the purification stage of the process for the production of lactic acid or of its derivatives.